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54 **Low iron loss grain oriented silicon steel sheets and method of producing the same.**

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Description

This invention relates to a method of producing low iron loss grain oriented silicon steel sheets.

5 Grain oriented silicon steel sheets are manufactured by methods involving many complicated steps requiring severe controls whereby the secondary recrystallized grains are highly aligned in the Goss orientation. Then a forsterite layer is formed on a surface of the base metal of the sheet and further an insulative layer having a small thermal expansion coefficient is formed thereon.

10 Such grain oriented silicon steel sheets are mainly used as cores for transformers and other electrical machinery and equipment. In this case, the magnetic properties of the sheet need to be such that the magnetic flux density (represented by the B_{10} value) is high and the iron loss (represented by the $W_{17/50}$ value) is low and the insulative layer needs to have good surface properties.

The need to reduce power loss has become more important in view of energy-saving, and hence it is necessary to provide grain oriented silicon steel sheets having a lower iron loss for use as transformer cores.

15 It is no exaggeration to say that the history of reducing the iron loss of grain oriented silicon steel sheets has depended on improving the secondary recrystallization structure of Goss orientation. As a method of controlling such a secondary recrystallized grain, there is practised a method of preferentially growing the secondary recrystallized grains of Goss orientation by using an agent for controlling the growth of the primary crystallized grains such as AlN, MnS, MnSe or the like, or a so-called inhibitor.

20 On the other hand, there have been proposed epoch-making techniques which are quite different from the above method of controlling the secondary recrystallization structure and wherein local microstrains are introduced by irradiating lasers onto the steel sheet surface (see T. Ichiyama: Tetsu To Hagane, 69(1983), p895, Japanese Patent Application Publication No. 57-2252, No. 57-53419, No. 58-24605 and No. 62-24606) or by plasma irradiation (see Japanese Patent laid open No. 62-96617, No. 62-151511, No. 62-151516 and
25 No. 62-151517) to refine the magnetic domains and thereby reduce the iron loss. In the steel obtained by these methods, however, the microstrain disappears when the sheets are heated up to a high temperature region. Thus these sheets can not be used for wound-core type transformers which are subjected to strain relief annealing at high temperature.

30 Furthermore, methods have been proposed in which there is no degradation of the iron loss property even when the sheet is subjected to strain relief annealing at high temperature. For example, there is a method involving forming a groove or serration on a surface of the finish annealed sheet (see Japanese Patent Application Publication No. 50-35679 and Japanese Patent laid open No. 59-28525 and No. 59-197520), a method involving producing fine regions of recrystallized grains on the surface of the finished annealed sheet (see Japanese Patent laid open No. 56-130454), a method involving forming different
35 thickness regions or deficient regions in the forsterite layer (see Japanese Patent laid open No. 60-92479, No. 60-92480, No. 60-92481 and No. 60-258479), a method involving forming different composition regions in the base metal, forsterite layer or tension insulative layer (Japanese Patent laid open No. 60-103124 and No. 60-103182), and the like.

40 In these methods, however, the steps become complicated, the effect of reducing the iron loss is less, and the production cost is high, so that such methods are not yet adopted industrially.

It is, therefore, an object of the invention to provide a method of stably producing low iron loss grain oriented silicon steel sheets by magnetic domain refinement which do not exhibit degradation of their iron loss properties on strain relief annealing.

45 According to the present invention there is provided a method of producing a low iron loss grain oriented silicon steel sheet, which comprises locally irradiating an electron beam on to a surface of a grain oriented silicon steel sheet, which has a surface layer after finish annealing, in a direction substantially perpendicular to the rolling direction of the sheet by dynamic focusing, whereby microareas of said surface layer are pushed into the base metal of the sheet at the positions irradiated by the electron beam.

50 The term "grain oriented silicon steel sheet which has a surface layer after finish annealing" used herein means a silicon steel sheet obtained by heating and hot rolling a silicon steel slab to form a hot rolled sheet, subjecting the hot rolled sheet to cold rolling twice with an intermediate annealing to form a final cold rolled sheet, subjecting the cold rolled sheet to decarburization and primary recrystallization annealing, applying a slurry of an annealing separator consisting mainly of MgO, and then subjecting the sheet to secondary recrystallization annealing for the preferential growth of secondary recrystallized grains
55 in the Goss orientation and purification annealing. Moreover, the term "finish annealing" means a combination of the secondary recrystallization annealing step and the purification annealing step. By "dynamic focusing" there is meant a technique where the irradiation by the electron beam is carried out by correcting the focusing distance of the electron beam at a proper distance so it is always focused at the surface of the

sheet in accordance with the change in distance between the electromagnetic lens and the sheet surface during the scanning of the electron beam.

Advantageously, the microareas extend from the front surface of the sheet through the base metal to the surface layer located at the rear surface of the sheet. In the latter case, micro-convex areas are formed on the rear surface of the sheet at a position corresponding to the pushed area of the front surface of the sheet.

In a preferred embodiment of the invention, the refinement of the magnetic domains is promoted by varying the irradiation diameter and the irradiation time of the electron beam to narrow the interval between the pushed microareas.

For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, in which:-

Figs. 1a and 1b are diagrammatic views showing the mechanism involved in the improvement of magnetic properties according to the invention,

Fig. 2 is a diagrammatic view showing the permeation force in the widthwise direction when silicon steel sheets are treated by various techniques;

Figs. 3a, 4a and 5a are schematic views showing electron beam (EB) irradiated tracks on a silicon steel sheets;

Figs. 3b, 4b and 5b are views corresponding to Figures 3a, 4a and 5a and showing the intensity of the EB;

Fig. 6 is a diagrammatic view of an EB irradiation apparatus for use in carrying out the invention;

Fig. 7a is a schematic view showing EB irradiated tracks on the sheet surface; and

Figs. 7b and 7c are views showing the intensity of the EB in the widthwise direction of the sheet during the scanning of the EB by different methods.

The invention will be described by reference to the experimental work carried out in the course of making the invention.

A slab of silicon steel containing C: 0.043% by weight (hereinafter referred to as % simply), Si: 3.45%, Mn: 0.068%, Se: 0.022%, Sb: 0.025% and Mo: 0.013% was heated at 1380 °C for 4 hours and hot rolled to form a hot rolled sheet of 2.2 mm in thickness. This sheet was then cold rolled twice with an intermediate annealing at 980 °C for 120 minutes to obtain a final cold rolled sheet of 0.20 mm in thickness. Next, the cold rolled sheet was subjected to decarburization and primary recrystallization annealing in a wet hydrogen atmosphere at 820 °C, coated with a slurry of an annealing separator consisting mainly of MgO, subjected to secondary recrystallization annealing at 850 °C for 50 hours to preferentially grow the secondary recrystallized grains in the Goss orientation and then subjected to purification annealing at 1200 °C in a dry hydrogen atmosphere for 5 hours to obtain sample sheet (A). Furthermore, an insulative layer consisting mainly of phosphate and colloidal silica was formed on a part of the sample sheet (A) to obtain sample sheet (B). Thereafter, the following treatments (1)-(4) were applied to each of the sample sheets (A) and (B), whereby microstrains or microareas were locally produced in a direction perpendicular to the rolling direction of the sheet at an interval of 8 mm.

(1) cutting with a knife;

(2) YAG laser irradiation (energy per spot: 4×10^{-3} J, spot diameter: 0.15 mm, distance between spot centres: 0.3 mm, scanning interval: 8 mm);

(3) EB irradiation (acceleration voltage: 100 kV, current: 0.7 mA, spot diameter: 1.0 mm, distance between spot centres: 0.3 mm, scanning interval: 8 mm);

(4) EB irradiation (acceleration voltage: 100 kV, current: 3.0 mA, spot diameter: 0.15 mm, distance between spot centres: 0.3 mm, scanning interval: 8 mm).

Each of the above treated samples was subjected to strain relief annealing at 800 °C for 2 hours. The magnetic properties measured after the strain relief annealing are shown in the following Table 1.

For comparison, the magnetic properties of a non-treated sheet (no introduction of microareas, strain relief annealing) are also shown in Table 1.

Table 1

Treatment	(A) Finish annealed sheet	(B) Formation of insulative layer on finish annealed sheet	Magnetic properties	
			B ₁₀ (T)	W _{17/50} (W/kg)
(1)	○	-	1.92	0.87
	-	○	1.91	0.86
(2)	○	-	1.92	0.85
	-	○	1.91	0.84
(3)	○	-	1.92	0.80
	-	○	1.92	0.79
(4)	○	-	1.92	0.79
	-	○	1.91	0.78
Comparative sheet	○	-	1.92	0.85
	-	○	1.91	0.86

As seen from Table 1, when each of the sample sheets (A) and (B) was subjected to each of the treatments (3) and (4), the iron loss value was improved by 0.05-0.08 W/kg as compared with those of the other cases.

In the sample sheets treated by the treatment (4), micro-convex areas were observed at the rear surface of the sheet, from which it is understood that the pushed microareas are introduced up to the rear surface of the sheet.

The reason why the iron loss value of the sample treated by the treatment (3) was improved as compared with those treated by the treatments (1) and (2) is due to the fact that as shown in Fig. 1a, microareas of forsterite layer 1 and insulative layer 2 pushed into base metal 3 (secondary recrystallised grains having a Goss orientation) in the depthwise direction thereof act as a nucleus for effective refinement of the magnetic domains even when subjected to strain relief annealing, whereby the magnetic domain refinement is made possible.

Further, the reason why the iron loss value of the sample treated by the treatment (4) was considerably improved as compared with those of the other samples is due to the fact that as shown in Fig. 1b, the pushed microareas further penetrate into the base metal 3 to extend up to the rear surface of the sheet, and act as strong nuclei for the magnetic domain refinement.

Moreover, the deep penetration of the microareas of the forsterite layer and insulative layer into the inside of the base metal in the widthwise direction of the sheet can be best achieved by using EB having a high voltage of 65-500 kV and a low current of 0.001-5 mA. As shown in Fig. 2, the use of high voltage and low current EB results in a permeation force which is strong in the depthwise direction and weak in the width direction as compared with the other means (laser, plasma, mechanical means and the like), so that the forsterite layer and the insulative layer can be pushed into the base metal without disappearance.

The EB irradiating conditions will now be described with respect to the following experiment.

A slab of silicon steel containing C: 0.042%, Si: 3.42%, Mn: 0.072%, Se: 0.021%, Sb: 0.023% and Mo: 0.013% was heated at 1370 °C for 4 hours and hot rolled to form a hot rolled sheet of 2.2 mm in thickness.

This was then cold rolled twice with an intermediate annealing at 980 °C for 120 minutes to obtain a final cold rolled sheet of 0.20 mm in thickness. After the cold rolled sheet was subjected to decarburization and primary recrystallisation annealing at 820 °C in a wet hydrogen atmosphere, a slurry of an annealing separator consisting mainly of MgO was applied to the sheet surface and then the sheet was subjected to

secondary recrystallization annealing at 850 °C for 50 hours to preferentially grow the secondary recrystallised grain in the Goss orientation and then subjected to purification annealing at 1200 °C in a dry hydrogen atmosphere for 5 hours to obtain sample sheet (C). Furthermore, an insulative layer consisting mainly of phosphate and colloidal silica was formed on a part of the sample sheet (C) to obtain sample sheet (D).

5 Thereafter, the following EB irradiation treatments (1)-(3) were applied to each of the sample sheets (C) and (D), whereby microareas were locally produced in a direction perpendicular to the rolling direction of the sheet at an interval of 8 mm.

(1) EB irradiation (acceleration voltage: 150 kV, current: 1.5 mA, spot diameter: 0.12 mm, distance between spot centres: 0.3 mm, scanning interval: 8 mm);

10 During the EB irradiation on to the steel sheet surface, the irradiated diameter of each spot and the irradiated distance between the spots were made uniform as shown in Fig. 3a. Fig. 3b shows the intensity of EB at each spot represented as the height of a triangle.

(2) EB irradiation (acceleration voltage: 150 kV, current: 1.5 mA or 0.75 mA, spot diameter: 0.12 mm or 0.80 mm, distance between spot centres: 0.3 mm, scanning interval: 8 mm).

15 During the EB irradiation on to the steel sheet surface, irradiated tracks as shown in Fig. 4a were formed by alternately changing the current between 1.5 mA and 0.75 mA and changing the irradiated diameter and the irradiated distance. Fig. 4b shows the intensity of EB represented as the height of a triangle as in Fig. 3b. As can be seen from the Figures, in this case, the sheet includes a plurality of larger microareas and a plurality of smaller microareas, wherein each larger microarea is adjacent to a smaller microarea to form a plurality of pairs of adjacent microareas, the microareas of each pair are arranged on opposite sides of an imaginary line extending in a direction substantially perpendicular to the rolling direction, the larger microareas of each adjacent pair of microareas are on opposite sides of the line, and the two microareas of each pair lie along a line inclined to the rolling direction.

20 (3) EB irradiation (acceleration voltage: 150 kV, current: 1.5 mA or 0.75 mA, spot diameter: 0.12 mm or 0.80 mm, distance between spot centres: 0.3 mm, scanning interval: 8 mm);

25 During the EB irradiation on to the steel sheet surface, irradiated tracks as shown in Fig. 5a were formed by changing the irradiated diameter and the irradiated distance and using currents of 1.5 mA and 0.75 mA. Fig. 5b shows the intensity of EB represented as the height of a triangle as in Fig. 3b. As can be seen from the Figures, in this case, the sheet includes a plurality of larger microareas and a plurality of smaller microareas which are arranged to form triplets of microareas each constituted by two of the larger microareas and one of the smaller microareas, the triplets being arranged along an imaginary line extending in a direction substantially perpendicular to the rolling direction with the larger microareas of each triplet being located on opposite sides of the line and the three microareas of each triplet lying along a line inclined to the rolling direction.

30 Each of the above treated samples was subjected to strain relief annealing at 800 °C for 2 hours. The magnetic properties measured after the strain relief annealing are shown in the following Table 2.

35 For comparison, the magnetic properties of a non-treated sheet (no introduction of microareas, strain relief annealing) are also shown in Table 2.

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Table 2

Treatment	(C) Finish annealed sheet	(D) Formation of insulative layer on finish annealed sheet	Magnetic properties		Lamination factor (%)
			B ₁₀ (T)	W _{17/50} (W/kg)	
(1)	○	-	1.92	0.82	96.6
	-	○	1.91	0.83	96.7
(2)	○	-	1.92	0.78	96.7
	-	○	1.91	0.79	96.8
(3)	○	-	1.92	0.77	96.7
	-	○	1.91	0.78	96.8
Comparative sheet	○	-	1.92	0.88	96.7
	-	○	1.91	0.89	96.8

As seen from Table 2, in the sample sheets (C) and (D) treated by EB, the iron loss value was improved by 0.05-0.11 W/kg as compared with those of the comparative sheet. Particularly, the iron loss value in case of the EB irradiation treatments (2) and (3) was largely improved by 0.10-0.11 W/kg. Furthermore, the products had a good lamination factor of 96.6-96.8%.

Further, it has been found that the permeation force of EB in the thickness direction (depthwise direction) of the silicon steel sheet increases at an acceleration voltage of not less than 65 kV usually generating a great amount of X-ray. In general, the acceleration voltage usually used for welding is not more than 60 kV, so that the permeation force is very small. That is, the above effect occurring during the method of the invention does not occur at such a conventional acceleration voltage. In order to utilize the effect of the invention to a maximum, therefore, it is important to set the acceleration voltage to a high value (65-500 kV) and the acceleration current to a small value (0.001-5 mA), whereby the permeation force in the thickness direction of the silicon steel sheet can be increased without causing breakage of the forsterite layer and insulative layer. Further, in order to efficiently conduct the magnetic domain refinement, the diameter of the irradiated area is preferably 0.005-0.3 mm by using a fine EB. Also, the direction of the scanning EB is substantially perpendicular to the rolling direction of the sheet, preferably at an angle of 60-90° with respect to the rolling direction. Preferably the distance between spot centres is 0.005-0.5 mm, the scanning interval is 2-20 mm, and the irradiation time per spot is 5-500 μsec. Moreover, the insulating property on the EB irradiated tracks may be enhanced by forming an insulative layer after the EB irradiation, but in this case the cost is increased. In general, a satisfactory insulating effect can be developed without the formation of an insulative layer after the EB irradiation.

The silicon steel sheets according to the invention may be used in the production of stacked lamination-core type transformers and wound-core type transformers as previously mentioned. In the case of stacked lamination-core type transformers, the introduction of microareas having a smaller spot diameter is required as compared with wound-core type transformers. For this purpose, the EB irradiating conditions should include a small current and a wide scanning interval. In the case of wound-core type transformers, it is preferred for the EB irradiating conditions to involve a somewhat large current and a narrow scanning interval for promoting the introduction of the microareas. Moreover, the EB may be irradiated on to one surface or on to both surfaces of the silicon steel sheet.

In Fig. 6 is schematically shown a preferred form of EB irradiation apparatus suitable for practising the invention, wherein 11 is a high voltage insulator, 12 is an EB gun, 13 is an anode, 14 is a column valve, 15 is an electromagnetic lens, 16 is a deflecting coil, 17 is an EB, 18 is a grain oriented silicon steel sheet and 19 and 20 are discharge ports, respectively.

In general, the EB irradiation on to the steel sheet surface is carried out in a direction substantially perpendicular to the rolling direction of the sheet as shown in Fig. 7a. In this case, since the current of the electromagnetic lens (focusing current) is constant, when the electromagnetic lens is focused at the centre of the sheet in the widthwise direction, the EB intensity is strongest at the central portion (17-2) of the sheet in the widthwise direction thereof and becomes weak at both end portions (17-1, 17-3) of the sheet as shown in Fig. 7b because the microareas are pushed into the sheet most effectively in positions where the EB is focused on to the steel sheet surface.

In accordance with the invention, the focusing distance of the EB is corrected in accordance with the change in distance between the electromagnetic lens and the sheet during the EB scanning so that the EB meets the sheet surface at a focusing position over the widthwise direction thereof. Such a correction of the focusing distance can be accurately carried out by dynamically controlling the currents of the electromagnetic lens 15 and the deflecting coil 16 shown in Fig. 6, whereby the EB scanning can be conducted at the same EB intensity over the full width of the sheet as shown in Fig. 7c. Such a treatment is referred to as dynamic focusing.

In this connection, the invention will be described with respect to the following experiment.

A slab of silicon steel containing C: 0.043%, Si: 3.39%, Mn: 0.066%, Se: 0.020%, Sb: 0.023% and Mo: 0.015% was heated at 1360 °C for 4 hours and hot rolled to form a hot rolled sheet of 2.0 mm in thickness. This was then subjected to a normalized annealing at 950 °C for 3 minutes and further cold rolled twice with an intermediate annealing at 950 °C for 3 minutes to obtain a final cold rolled sheet of 0.20 mm in thickness.

After the cold rolled sheet was subjected to decarburization and primary recrystallization annealing at 820 °C in a wet hydrogen atmosphere, a slurry of an annealing separator consisting mainly of MgO was applied to the sheet surface, and then the sheet was subjected to finish annealing.

After an insulative layer consisting mainly of phosphate and colloidal silica was formed on the sheet surface, the sheet was subjected to EB irradiation without dynamic focusing (a-1) or EB irradiation with dynamic focusing (a-2). For comparison, there was provided a sheet not subjected to EB irradiation (a-3).

In another experiment a slurry of annealing separator consisting mainly of Al₂O₃ was applied to the sheet surface after the above primary recrystallization annealing and the sheet subjected to finish annealing under the same conditions as mentioned above. Thereafter, the finished annealed sheet was lightly pickled and subjected to an electrolytic polishing to provide it with a mirror surface having a centre-line average roughness of Ra = 0.1 μm, on which a thin layer of TiN having a thickness of 1.0 μm was formed by an ion plating apparatus by an HCD method (acceleration voltage: 70 V, acceleration current: 1000 A, vacuum degree: 7x10⁻⁴ Torr = 9.33 x 10⁻⁴ mbar)

A part of the sheet was then subjected to EB irradiation without dynamic focusing (b-1) or EB irradiation with dynamic focusing (b-2) and an insulative layer consisting mainly of phosphate and colloidal silica was formed thereon.

An insulative layer consisting mainly of phosphate and colloidal silica was formed on another part of the sheet provided with the thin TiN layer, which was then subjected to EB irradiation without dynamic focusing (b-3) or EB irradiation with dynamic focusing (b-4).

For comparison, there was provided a sheet provided with the insulative layer but not subjected to EB irradiation treatment (b-5).

The magnetic properties of each of the thus obtained products are shown in the following Table 3.

Table 3

Treatment	Sample	EB irradiation method	Magnetic properties	
			B ₁₀ (T)	W _{17/50} (W/kg)
a-1	Finish annealed sheet	① usual EB irradiation *	1.90	0.82
a-2		② EB irradiation through dynamic focusing **	1.91	0.78
a-3		③ ———	1.90	0.85
b-1	Sheet provided at its surface with TiN layer after mirror polishing of finish annealed sheet	① usual EB irradiation *	1.92	0.66
b-2		② EB irradiation through dynamic focusing **	1.93	0.63
b-3		① usual EB irradiation *	1.92	0.67
b-4		② EB irradiation through dynamic focusing **	1.93	0.64
b-5		③ ———	1.92	0.70

* ① usual EB irradiation : acceleration voltage: 70 kV,
acceleration current: 7 mA,
scanning interval in a direction
perpendicular to rolling direction:
300 μm, scanning width: 10 mm.

** ② EB irradiation through
dynamic focusing: acceleration voltage: 70 kV,
acceleration current: 7 mA,
scanning interval in a direction
perpendicular to rolling direction:
300 μm, scanning width: 10 mm,
dynamic focusing of electromagnetic
lens and deflecting coil.

As can be seen from Table 3, when the sheet is subjected to EB irradiation with dynamic focusing, the iron loss property is further improved as compared with the case where no dynamic focusing is used.

Thus, a further reduction of iron loss can be attained by adopting dynamic focusing in the widthwise direction of the sheet when a grain oriented silicon steel sheet provided with an insulative layer after finish annealing is subjected to EB irradiation or when a sheet provided with a TiN layer after the mirror polishing of the finish annealed sheet is subjected to EB irradiation before or after the formation of an insulative layer. That is, in the case of dynamic focusing, the focusing distance of the electron beam is corrected so that it is always located at the sheet surface irrespective of the change of the focusing position during EB scanning as shown in Fig. 7c. In this way constant irradiated tracks are formed over the widthwise direction of the sheet to effectively conduct the refinement of the magnetic domains over the whole area of the sheet, and consequently low iron loss silicon steel sheets can be obtained.

The following Examples are given to illustrate the invention and are not intended as limitations thereof.

Example 1

5 A slab of (A) silicon steel containing C: 0.043%, Si: 3.36%, Se: 0.02%, Sb: 0.025% and Mo: 0.013% and a slab of (B) silicon steel containing C: 0.063%, Si: 3.42%, Al: 0.025%, S: 0.023%, Cu: 0.05% and Sn: 0.1% were each heated at 1380 °C for 4 hours and hot rolled to obtain hot rolled sheets of 2.2 mm in thickness. These were then cold rolled twice with an intermediate annealing at 980 °C for 120 minutes to
10 obtain final cold rolled sheets of 0.20 mm in thickness. After the cold rolled sheets were subjected to decarburization and primary recrystallization annealing at 820 °C in a wet hydrogen atmosphere, a slurry of an annealing separator consisting mainly of MgO was applied to the surface of each sheet, which was then subjected to a finish annealing, wherein secondary recrystallization annealing was carried out at 850 °C for 50 hours to preferentially grow secondary recrystallized grains in the Goss orientation and purification annealing was carried out at 1200 °C in a dry hydrogen atmosphere for 5 hours, whereby finish annealed
15 sheets (thickness: 0.20 mm) provided with a forsterite layer were obtained. Further, a part of each sheet was provided at its surface with an insulative layer.

These sheets were subjected to EB irradiation in a direction perpendicular to the rolling direct of the sheet by means of an EB irradiation apparatus under conditions that the acceleration voltage was 100 kV, the acceleration current was 0.5 mA, the spot diameter was 0.1 mm, the distance between spot centres was
20 0.3 mm and the scanning interval was 8 mm, provided that the microareas pushed did not reach to the layers at the rear surface of the sheets.

After the sheets were subjected to strain relief annealing at 800 °C for 2 hours, the magnetic properties were measured and the results are shown in the following Table 4 together with those for a comparative sheet (no introduction of microareas, strain relief annealing). As can be seen from Table 4, the iron loss
25 $W_{17/50}$ was reduced by 0.08-0.1 W/kg as compared with that of the comparative sheet.

Table 4

Sample	Finish annealed	Insulative layer formed on finish annealed sheet	Magnetic properties		EB irradiation
			B_{10} (T)	$W_{17/50}$ (W/kg)	
(A)	○	-	1.92	0.79	irradiated
	-	○	1.91	0.77	
(B)	○	-	1.94	0.78	
	-	○	1.93	0.76	
Comparative sheet	○	-	1.92	0.86	not irradiated
	-	○	1.91	0.87	

Example 2

50 A slab of (A) silicon steel containing C: 0.042%, Si: 3.38%, Se: 0.023%, Sb: 0.026% and Mo: 0.012% and a slab of (B) silicon steel containing C: 0.061%, Si: 3.44%, Al: 0.026%, S: 0.028%, Cu: 0.08% and Sn: 0.15% were treated by the same manner as in Example 1 to obtain finish annealed sheets (thickness: 0.20 mm) provided with a forsterite layer. Further, a part of each sheet was provided at its surface with an insulative layer.

55 These sheets were subjected to EB irradiation according to the scanning pattern shown in Fig. 5 in a direction perpendicular to the rolling direction of the sheet by means of an EB irradiation apparatus under conditions that the acceleration voltage was 150 kV, the acceleration current was 1.5 mA, the spot diameter was 0.1 mm or 0.7 mm, the distance between spot centres was 0.3 mm and the scanning interval was 8

mm, provided that the microareas pushed reached to the layer at the rear surface of the sheets.

After the sheets were subjected to strain relief annealing at 800 °C for 2 hours, the magnetic properties were measured and the results are shown in the following Table 5 together with those for a comparative sheet (no introduction of microareas, strain relief annealing). As can be seen from Table 5, the iron loss $W_{17/50}$ was reduced by 0.10-0.14 W/kg as compared with that of the comparative sheet.

Table 5

Sample	Finish annealed	Insulative layer formed on finish annealed sheet	Magnetic properties		EB irradiation
			B_{10} (T)	$W_{17/50}$ (W/kg)	
(A)	○	-	1.92	0.78	irradiated
	-	○	1.91	0.76	
(B)	○	-	1.94	0.77	
	-	○	1.93	0.75	
Comparative sheet	○	-	1.92	0.88	not irradiated
	-	○	1.91	0.89	

Example 3

A slab of (A) silicon steel containing C: 0.040%, Si: 3.45%, Se: 0.025%, Sb: 0.030% and Mo: 0.015% and a slab of (B) silicon steel containing C: 0.057%, Si: 3.42%, sol Al: 0.026%, S: 0.029%, Cu: 0.1% and Sn: 0.050% were heated at 1380 °C for 4 hours and hot rolled to obtain hot rolled sheets of 2.2 mm in thickness. These were then cold rolled two times with an intermediate annealing at 1050 °C for 2 minutes to obtain final cold rolled sheets of 0.20 mm in thickness. After the cold rolled sheets were subjected to decarburization and primary recrystallization annealing at 840 °C in a wet hydrogen atmosphere, a slurry of (a) an annealing separator consisting mainly of MgO or (b) an annealing separator consisting of Al₂O₃: 60%, MgO: 35%, ZrO₂: 3% and TiO₂: 2% was applied to the surface of the sheets.

After the application of annealing separator (a), sheet (A) was subjected to secondary recrystallization annealing at 850 °C for 50 hours and further to purification annealing at 1200 °C in a dry hydrogen atmosphere for 5 hours, while sheet (B) was subjected to secondary recrystallization annealing by heating from 850 °C to 1050 °C at a rate of 10 °C/hr and further to purification annealing at 1220 °C in a dry hydrogen atmosphere for 8 hours.

Then, an insulative layer consisting mainly of phosphate and colloidal silica was formed on the surface of each of these sheets.

On the other hand, after the application of annealing separator (b), each sheet was pickled to remove oxides from the surface and subjected to electrolytic polishing into a mirror state, on which was formed a TiN tension layer of 1.0 μm in thickness by means of an ion plating apparatus and further the same insulative layer as mentioned above was formed thereon.

Thereafter, each of the sheets was subjected to EB irradiation with dynamic focusing by means of the apparatus shown in Fig. 6 at an interval of 8 mm in a direction perpendicular to the rolling direction of the sheet under conditions such that the acceleration voltage was 70 kV, the current was 10 mA and the scanning interval was 200 μm. Then, the magnetic properties were measured and the results (average values in the widthwise direction of the sheet) are shown in the following Table 6.

Table 6

Kind of steel	Annealing separator	Surface layer	Magnetic properties	
			B ₁₀ (T)	W _{17/50} (W/kg)
A	a	only insulative layer	1.91	0.78
	b	TiN + insulative layer	1.93	0.63
B	a	only insulative layer	1.93	0.79
	b	TiN + insulative layer	1.94	0.64

Claims

1. A method of producing a low iron loss grain oriented silicon steel sheet, which comprises locally irradiating an electron beam on to a surface of a grain oriented silicon steel sheet, which has a surface layer after finish annealing, in a direction substantially perpendicular to the rolling direction of the sheet by dynamic focusing, whereby microareas of said surface layer are pushed into the base metal of the sheet at the positions irradiated by the electron beam.
2. A method according to claim 1 wherein the electron beam is generated at an acceleration voltage of from 65 to 500 kV and an acceleration current of 0.001 to 5 mA.
3. A method according to claim 1 or 2 wherein the surface layer comprises a forsterite layer and an insulative layer
4. A method according to claim 1, 2 or 3 wherein said irradiating is effected so that said base metal is simultaneously pushed into the rear surface of said sheet to form micro convex areas at said positions.
5. A method according to any preceding claim wherein said electron beam is irradiated at a beam diameter of 0.005-0.3 mm and an irradiation time per spot of 5-500 μ sec so that said microareas are arranged in the form of spots having a diameter of 0.005-0.3 mm and the distance between spot centres is 0.005-0.5 mm at a scanning interval of the electron beam of 2-20 mm.
6. A method according to any preceding claim wherein the electron beam is generated using first and second acceleration currents alternatively so as to provide the sheet with pushed microareas of two different sizes.
7. A method according to claim 6 wherein the electron beam is such that the sheet includes a plurality of larger microareas and a plurality of smaller microareas wherein each larger microarea is adjacent to a smaller microarea to form a plurality of pairs of adjacent microareas, the microareas of each pair are arranged on opposite sides of an imaginary line extending in a direction substantially perpendicular to the rolling direction, the larger microareas are on opposite sides of the line, and the two microareas of each pair lie along a line inclined to the rolling direction.
8. A method according to claim 6 wherein the electron beam is such that the sheet includes a plurality of larger microareas and a plurality of smaller microareas which are arranged to form triplets of microareas each constituted by two of the larger microareas and one of the smaller microareas, the triplets being arranged along an imaginary line extending in a direction substantially perpendicular to the rolling direction with the larger microareas of each triplet being located on opposite sides of the line and the three microareas of each triplet lying along a line inclined to the rolling direction.

Patentansprüche

1. Verfahren zum Herstellen eines kornorientierten Siliziumstahlbleches mit niedrigen Eisenverlusten, bei welchem eine Fläche eines kornorientierten Siliziumstahlbleches, welches nach dem Fertigglühen eine

Oberflächenschicht aufweist, in einer im wesentlichen senkrecht zur Walzrichtung des Stahlblechs verlaufenden Richtung unter dynamischem Focussieren lokal mit einem Elektronenstrahl bestrahlt wird, womit Mikrobereiche der erwähnten Oberflächenschicht an durch den Elektronenstrahl beaufschlagten Stellen in das Grundmetall hineingedrückt werden.

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2. Verfahren nach Anspruch 1, worin der Elektronenstrahl bei einer Beschleunigungsspannung von 65 bis 500 kV und mit einem Strahlstrom von 0,001 bis 5 mA erzeugt wird.

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3. Verfahren nach Anspruch 1 oder 2, worin die Oberflächenschicht eine Forsteritschicht und eine Isolierschicht aufweist.

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4. Verfahren nach Anspruch 1, 2 oder 3, worin das Bestrahlen so durchgeführt wird, daß das Grundmetall gleichzeitig in die Rückseite des Bleches unter Ausbildung von konvexen Mikrobereichen an den erwähnten Stellen hineingedrückt wird.

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5. Verfahren nach irgendeinem vorhergehenden Anspruch, worin der Elektronenstrahl mit einem Strahldurchmesser von 0,005 bis 0,3 mm bei einer Bestrahlungszeit von 5 bis 500 μ s je Fleck in solcher Weise abgestrahlt wird, daß die erwähnten Mikrobereiche in Form von Flecken mit einem Durchmesser von 0,005 bis 0,3 mm mit einem Abstand von 0,005 bis 0,5 mm zwischen den Fleckmittelpunkten bei einem Abtastabstand des Elektronenstrahls von 2 bis 20 mm angeordnet werden.

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6. Verfahren nach irgendeinem vorhergehenden Anspruch, worin der Elektronenstrahl unter alternierender Verwendung eines ersten und eines zweiten Strahlstroms in solcher Weise erzeugt wird, daß das Blech mit hineingedrückten Mikrobereichen von zwei verschiedenen Abmessungen versehen wird.

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7. Verfahren nach Anspruch 6, worin der Elektronenstrahl ein solcher ist, daß das Blech mehrere größere Mikrobereiche und mehrere kleinere Mikrobereiche aufweist und hierbei jeder größere Mikrobereich einem benachbarten Mikrobereich unter Ausbildung mehrerer benachbarter Mikrobereiche benachbart ist, wobei die Mikrobereiche eines jeden Paares an gegenüberliegenden Seiten einer sich im wesentlichen senkrecht zur Walzrichtung erstreckenden gedachten Linie liegen und hierbei die größeren Mikrobereiche an gegenüberliegenden Seiten liegen und die Mikrobereiche eines jeden Paares entlang einer zur Walzrichtung geneigten Linie liegen.

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8. Verfahren nach Anspruch 6, worin der Elektronenstrahl ein solcher ist, daß das Blech mehrere größere Mikrobereiche und mehrere kleinere Mikrobereiche aufweist und hierbei die Mikrobereiche in Triplettts von Mikrobereichen angeordnet sind, wobei jedes Triplett von zwei der größeren Mikrobereiche und von einem der kleineren Mikrobereiche gebildet ist und die Triplettts entlang einer zur Walzrichtung im wesentlichen senkrecht verlaufenden Linie in solcher Weise angeordnet sind, daß die größeren Mikrobereiche eines jeden Triplettts an gegenüberliegenden Seiten dieser Linie gelegen sind und die drei Mikrobereiche eines jeden Triplettts entlang einer zur Walzrichtung geneigten Linie liegen.

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Revendications

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1. Un procédé de fabrication d'une tôle d'acier au silicium à grain orienté à faible perte dans le fer, qui consiste à effectuer une irradiation locale d'un faisceau d'électrons sur une surface d'une tôle d'acier au silicium à grain orienté, qui présente une couche de surface après recuit de fini, dans une direction sensiblement perpendiculaire à la direction de laminage de la tôle par focalisation dynamique, de sorte que des microzones de ladite couche de surface sont repoussées dans le métal de base de la tôle aux endroits irradiés par le faisceau d'électrons.

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2. Un procédé selon la revendication 1, dans lequel le faisceau d'électrons est engendré à une tension d'accélération de 65 à 500 kV et un courant d'accélération de 0,001 à 5 mA.

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3. Un procédé selon la revendication 1 ou 2, dans lequel la couche de surface comprend une couche de forstérite et une couche isolante.

4. Un procédé selon la revendication 1, 2 ou 3, dans lequel ladite irradiation est effectuée de façon que ledit métal de base soit simultanément repoussé dans la surface arrière de ladite tôle pour former des

zones microconvexes auxdits endroits.

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6. Un procédé selon l'une quelconque des revendications précédentes, dans lequel ledit faisceau d'électrons est irradié à un diamètre de faisceau de 0,005 à 0,3 mm et un temps d'irradiation par tache de 5 à 500 μ sec de façon que lesdites microzones soient disposées sous la forme de taches ayant un diamètre de 0,005 à 0,3 mm et que la distance entre les centres des taches soit de 0,005 à 0,5 mm à un intervalle de balayage du faisceau d'électrons de 2 à 20 mm.
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7. Un procédé selon l'une quelconque des revendications précédentes, dans lequel le faisceau d'électrons est engendré en utilisant un premier et un second courant d'accélération alternativement de façon à obtenir la tôle avec des microzones poussées de deux tailles différentes.
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8. Un procédé selon la revendication 6, dans lequel le faisceau d'électrons est tel que la tôle comprend une pluralité de microzones plus grandes et une pluralité de microzones plus petites dans laquelle chaque microzone plus grande est adjacente à une microzone plus petite pour former une pluralité de paires de microzones adjacentes, les microzones de chaque paire sont disposées sur les côtés opposés d'une ligne imaginaire s'étendant dans une direction sensiblement perpendiculaire à la direction de laminage, les microzones plus grandes sont sur les côtés opposés de la ligne, et les deux microzones de chaque paire s'étendent le long d'une ligne inclinée par rapport à la direction de laminage.
- 20
9. Un procédé selon la revendication 6, dans lequel le faisceau d'électrons est tel que la tôle comprend une pluralité de microzones plus grandes et une pluralité de microzones plus petites qui sont disposées pour former des triplés de microzones, chacune constituée par deux des microzones plus grandes et une des microzones plus petites, les triplés étant disposés le long d'une ligne imaginaire s'étendant dans une direction sensiblement perpendiculaire à la direction de laminage avec les microzones plus grandes de chaque triplé étant disposées sur les côtés opposés de la ligne et les trois microzones de chaque triplé s'étendant le long d'une ligne inclinée par rapport à la direction de laminage.
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- 30
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- 55

FIG. 1a

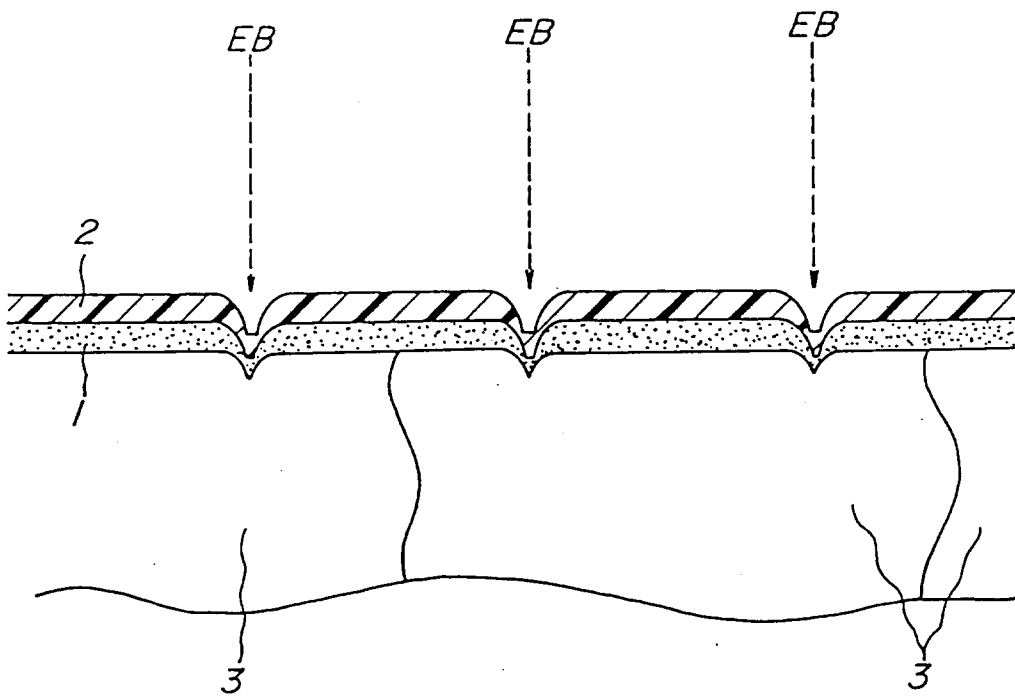


FIG. 1b

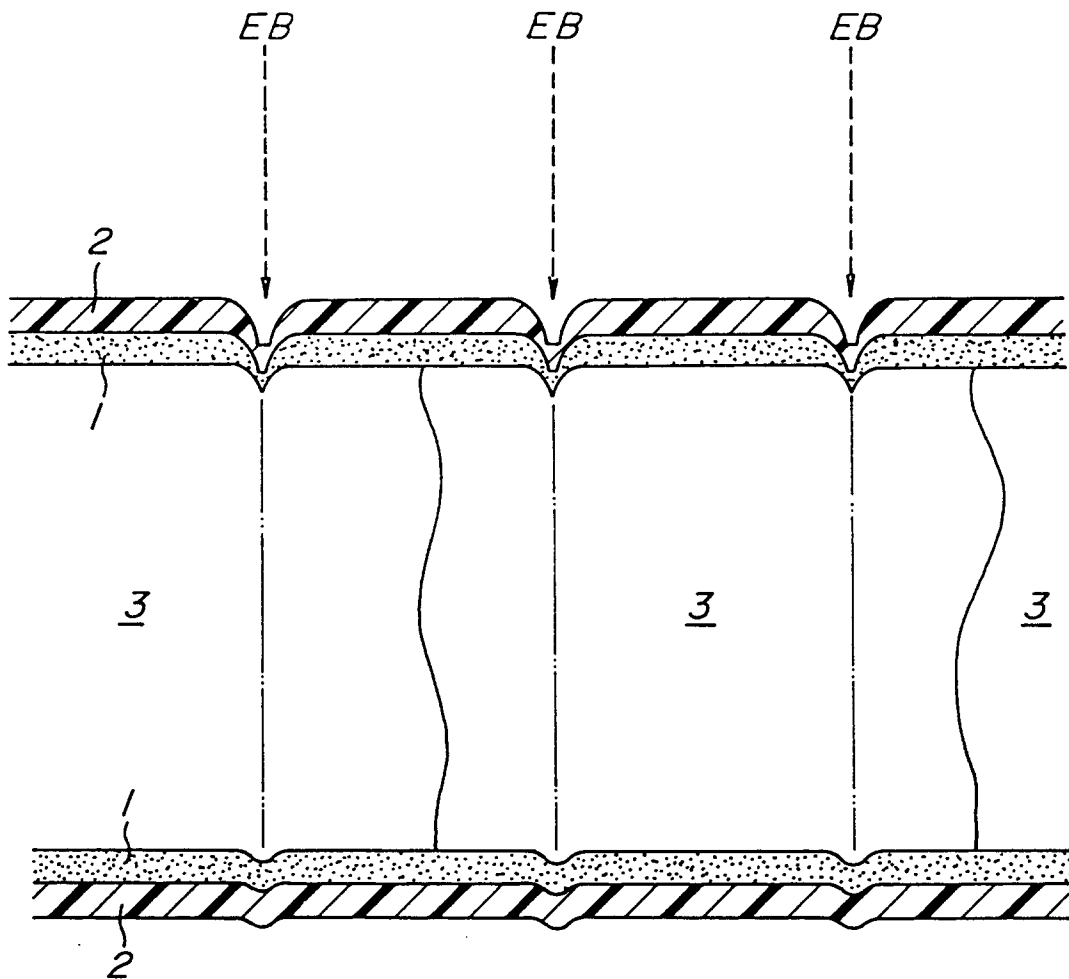


FIG. 2

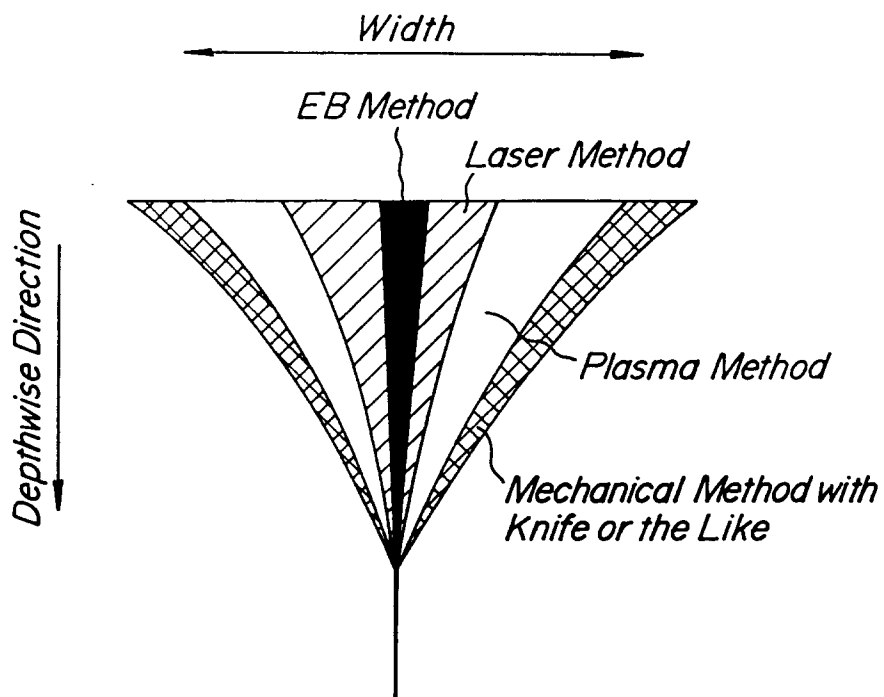


FIG. 3a

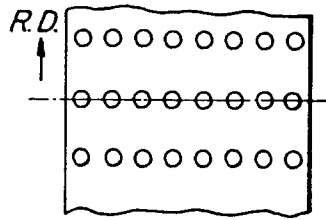


FIG. 3b



FIG. 4a

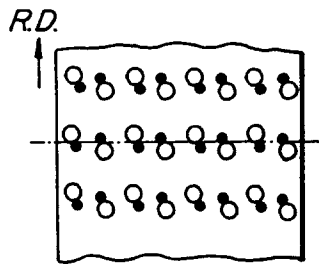


FIG. 4b



FIG. 5a

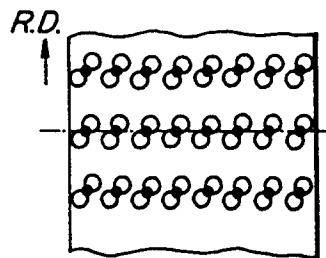


FIG. 5b



FIG. 6

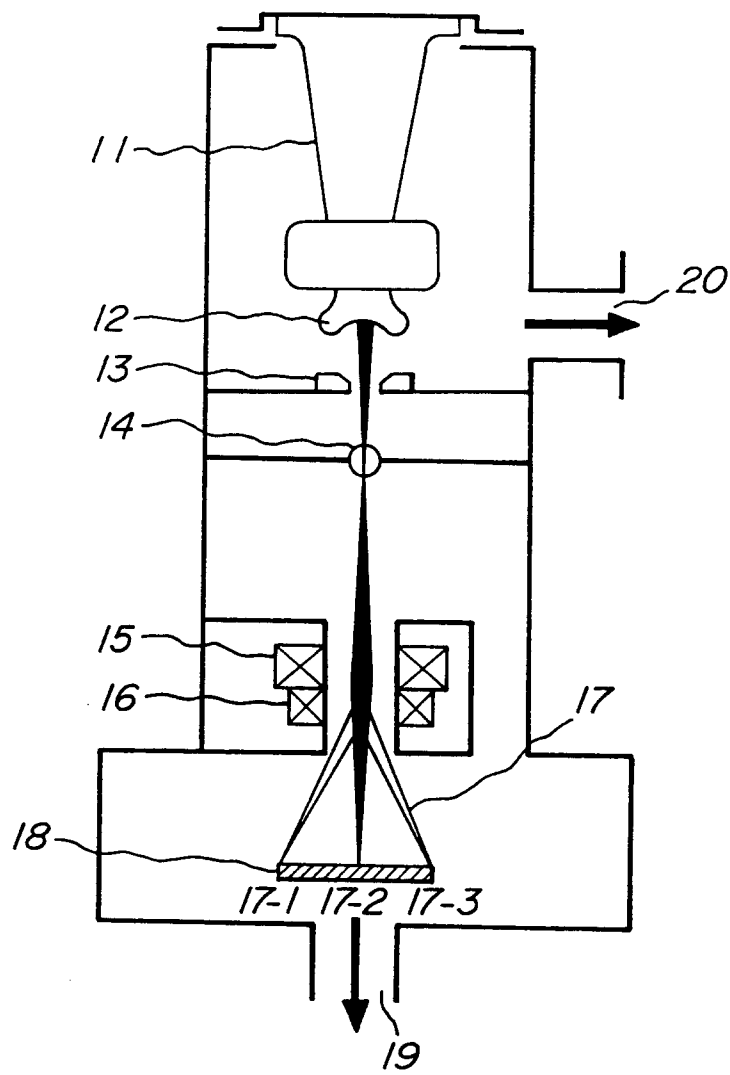


FIG. 7a

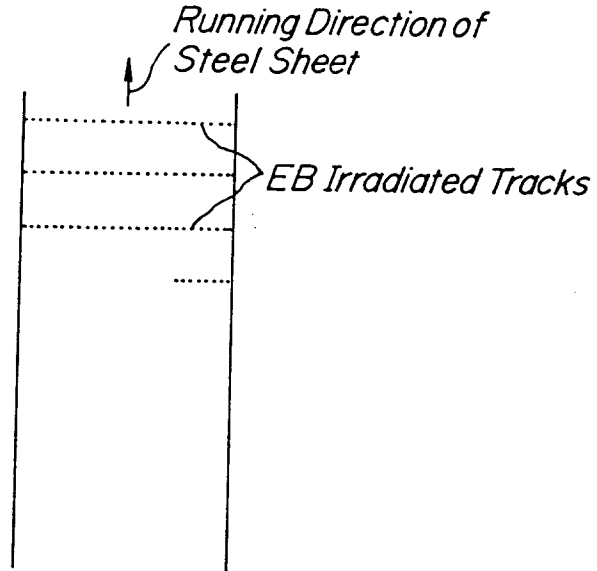


FIG. 7b

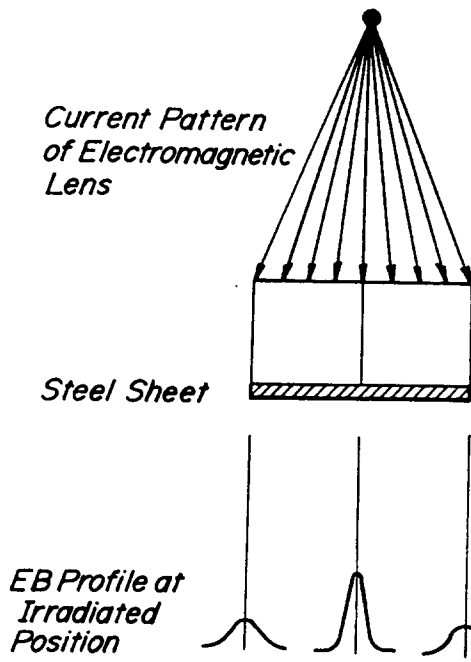


FIG. 7c

